Appendix J

ESEM and SEM/EDS Data for Test-2 Day-4 Filtrate and Fiberglass Samples

List of Figures

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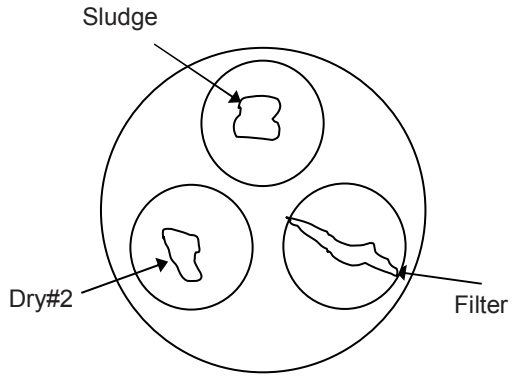
This appendix presents ESEM and SEM/EDS images for filtrate collected from a filtered solution sample and for sacrificial fiberglass samples removed from the test tank on Day 4 (February 9, 2005) of Test #2. The filtered solution sample was passed through a 0.7-μm fiberglass filter at 60°C. SEM/EDS results for the resulting filtrate are presented here. These SEM results were obtained on February 25, 2005.

The fiberglass samples were taken from material encased in a small (~4 in. × 4 in.) stainless steel mesh envelope that had been submerged in the test tank until being removed on Day 4. Fiberglass samples were examined both hydrated (its condition when removed from the tank) and dry (air dried at room temperature).

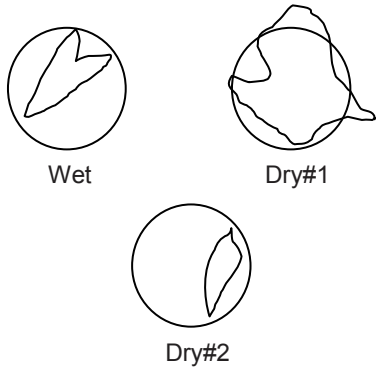
Transcribed Laboratory Log

Laboratory session from February 9, 2005

NRC Test 2 Day 4



SEM MicroProbe Sample Arrangement



ESEM Sample Arrangement

Instrument Conditions: ESEM work in low-vacuum mode with BSE imaging, 20-kV, Working Distance = 8 mm, Aperture = 2, low vacuum = 100 Pa (starting pressure)

Hydrated Sample

Image:	T2D4001	110 ×	Overview	Figure J-1
	T2D4002	600 ×	Close-up near center of image 001	Figure J-2
	T2D4003	500 ×	Close-up of film near center of image 001	Figure J-3
	T2D4004	110 ×	New area	Figure J-4
	T2D4005	500 ×	Right center of image 004	Figure J-5

Dry#1 Sample

Image:	T2D4006	120 ×	Overview	Figure J-6
	T2D4007	500 ×	On deposits between fibers	Figure J-7
	T2D4008	130 ×	Overview new area	Figure J-8
	T2D4009	500 ×	More deposits	Figure J-9

Dry#2 Sample

Image:	T2D4010	40 ×	Overview	Figure J-10
	T2D4011	250 ×	On deposits w/ crystals	Figure J-11
EDS:	D2-1		On smooth deposits	Figure J-12
	D2-2		On crystal mass on fibers	Figure J-13
	D2-3		On smooth cracked deposits	Figure J-14
Image:	T2D4012	200 ×	Crystals on fibers	Figure J-15
EDS:	D2-4		On mass of crystals on fiber, center of image 012	Figure J-16

Filter Sample

Image:	T2D4015	40 ×	Overview of filtrate surface	Figure J-17
	T2D4016	2000 ×	Close-up of filtrate	Figure J-18
EDS:	F-7		On filtrate	Figure J-19
	F-8		On filtrate	Figure J-20

Note: EDS F-7 and F-8 are replicates of homogeneous filtrate material.

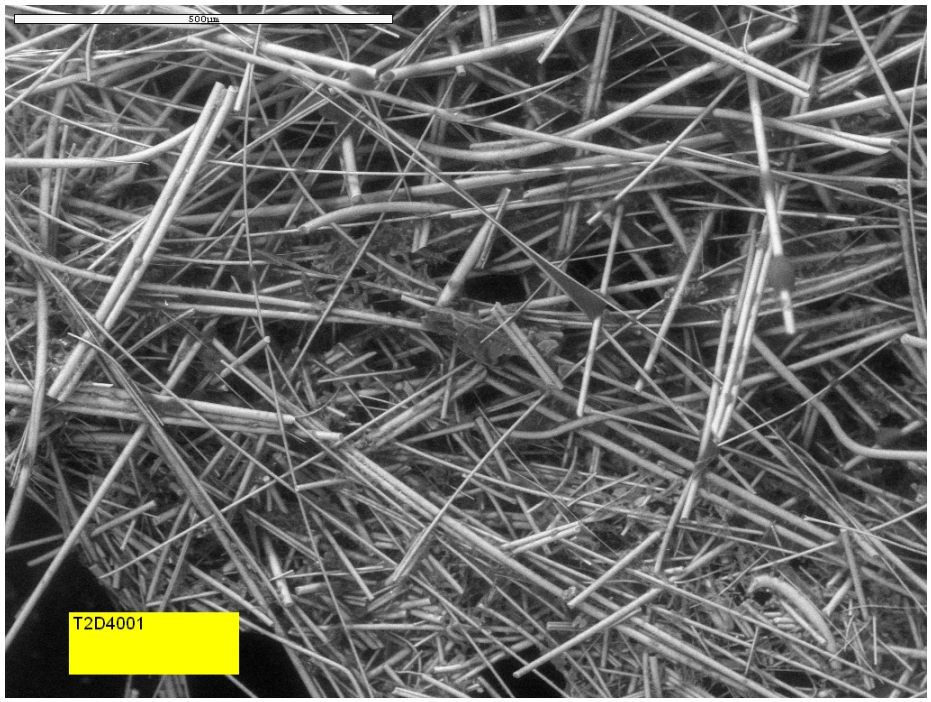


Figure J-1. Test-2 Day-4 ESEM image of a hydrated sample, overview (T2D4001).

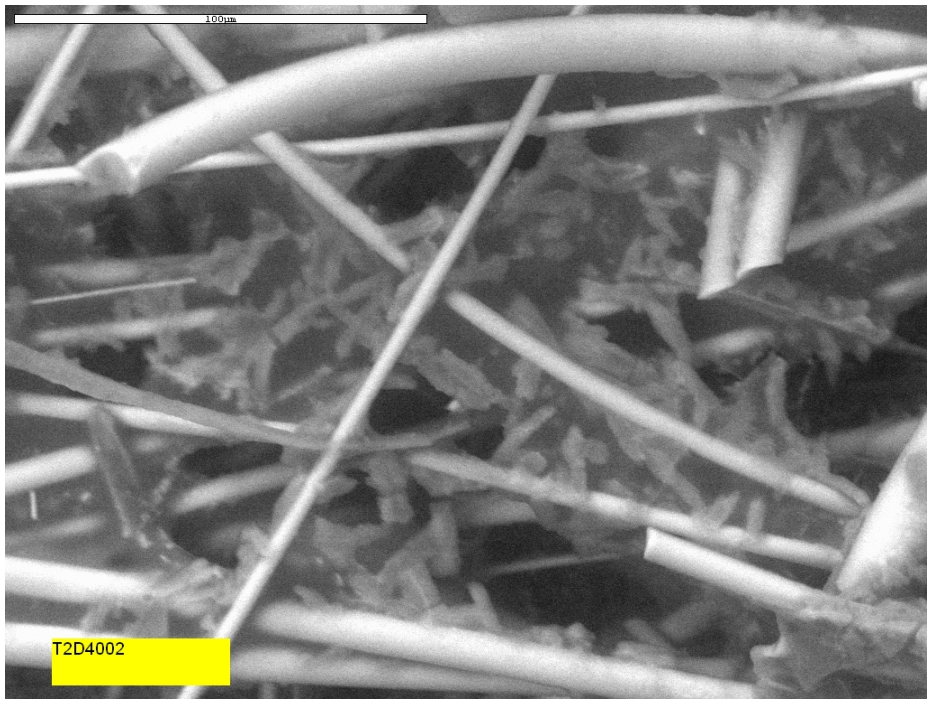


Figure J-2. Test-2 Day-4 ESEM image of a hydrated sample, close-up near the center of the image in Figure J-1.

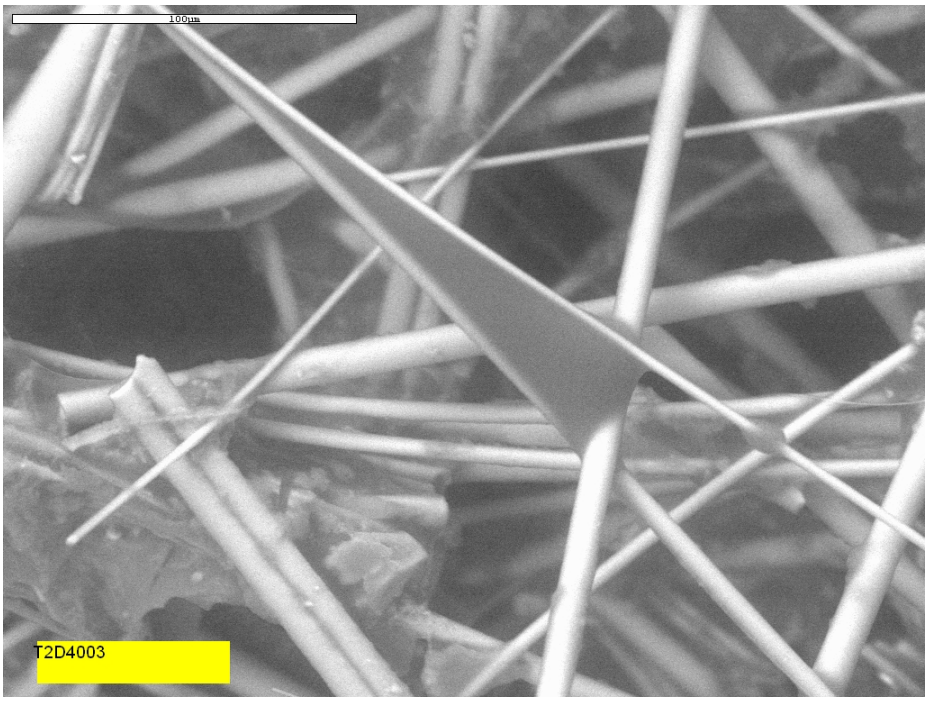


Figure J-3. Test-2 Day-4 ESEM image, hydrated sample close-up on the film near the center of the image in Figure J-1 (T2D4003).

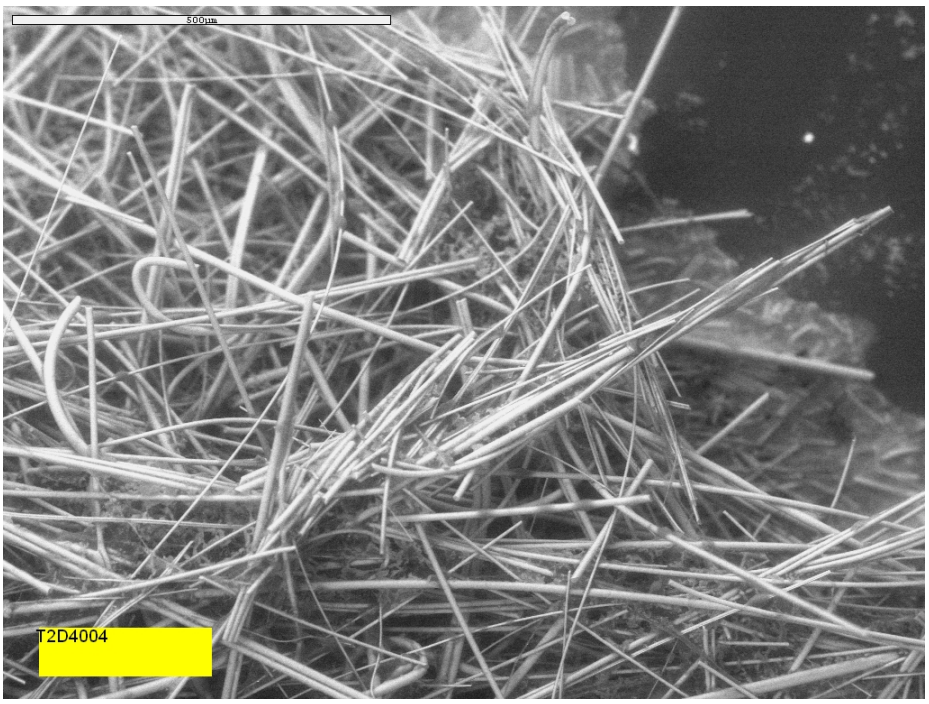


Figure J-4. Test-2 Day-4 ESEM image of a new area within the hydrated sample (T2D4004).

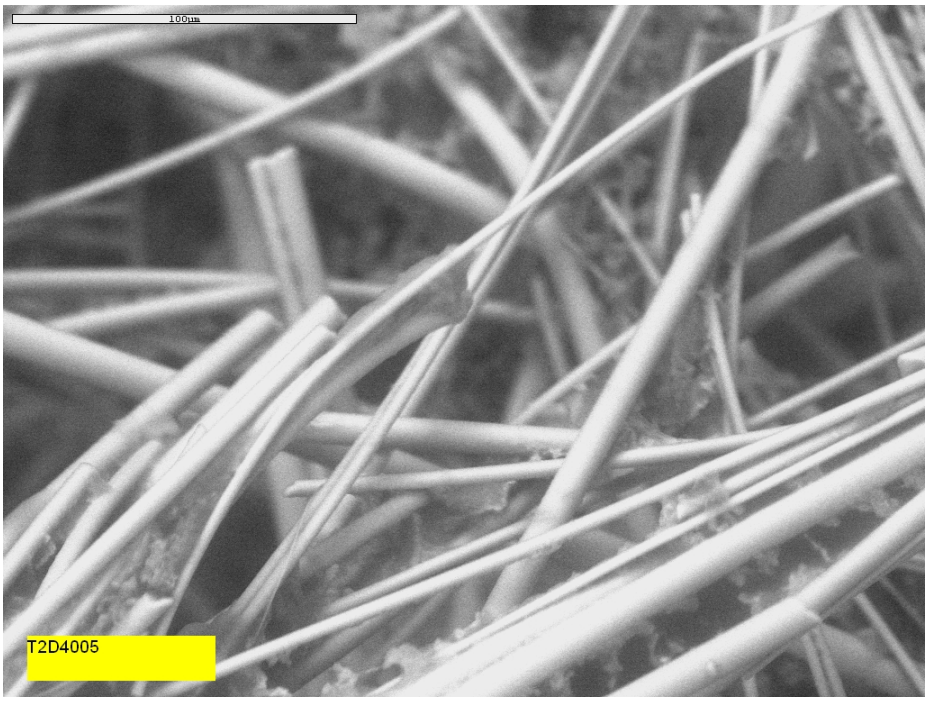


Figure J-5. Test-2 Day-4 ESEM image of the hydrated sample at the right center of Figure J-4 (T2D4005).

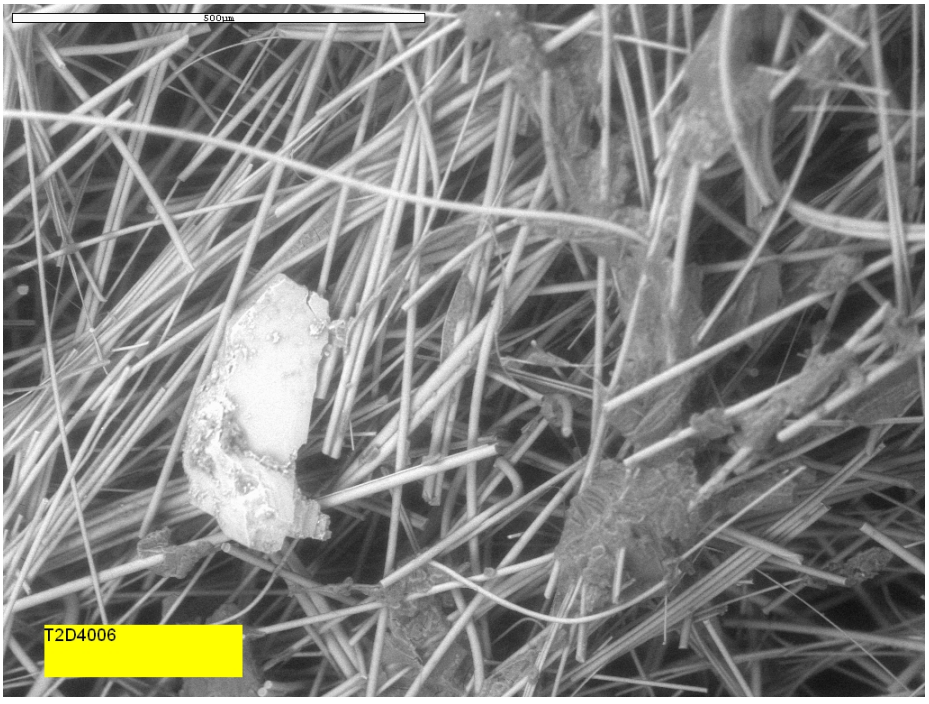


Figure J-6. Test-2 Day-4 SEM image of dry sample #1, overview (T2D4006).

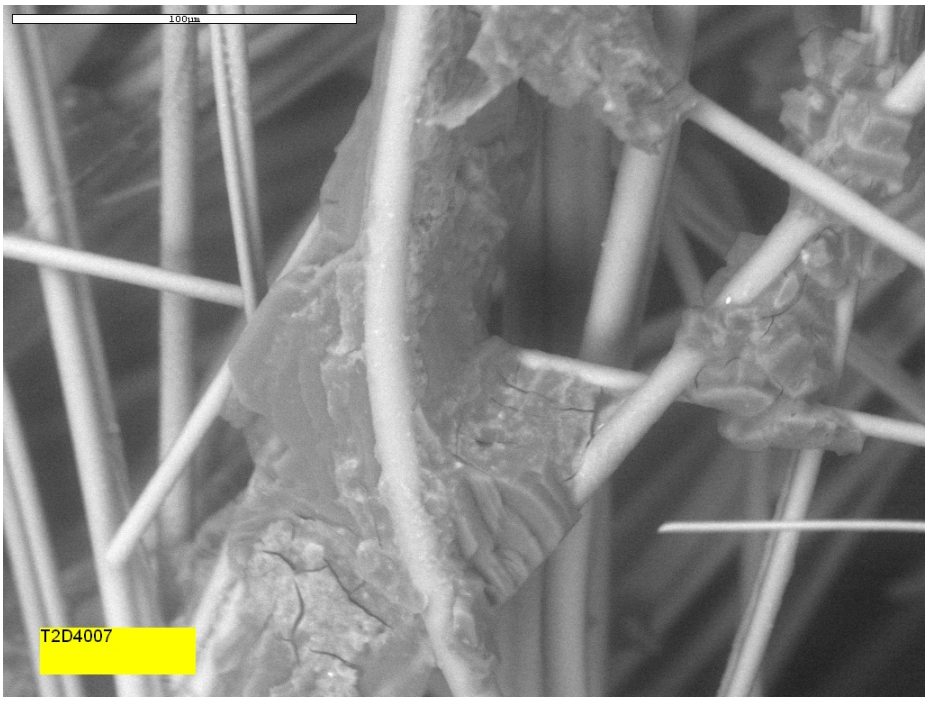


Figure J-7. Test-2 Day-4 SEM dry sample #1, image of deposits between fibers (T2D4007).

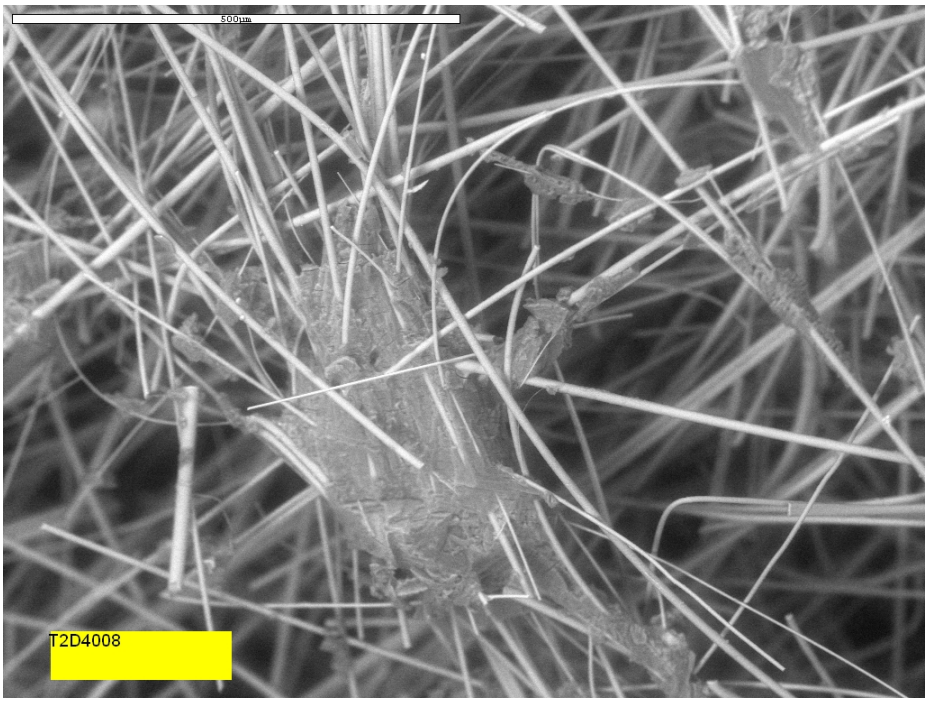


Figure J-8. Test-2 Day-4 SEM image, overview of a new area of dry sample #1 (T2D4008).

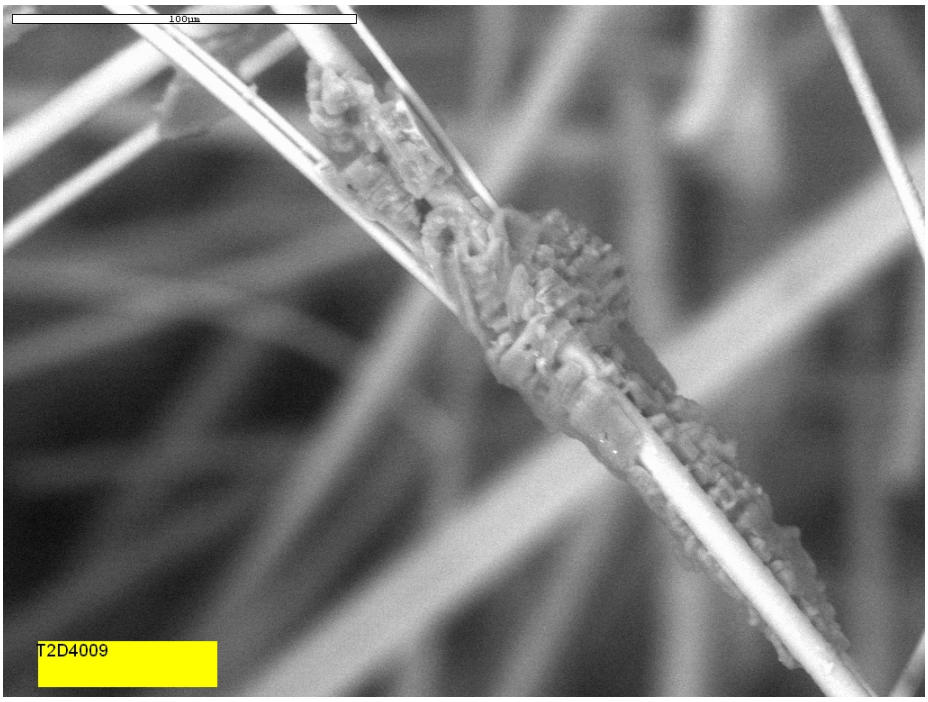


Figure J-9. Test-2 Day-4 SEM image of deposits on fibers of dry sample #1 at 500 × magnification (T2D4009).

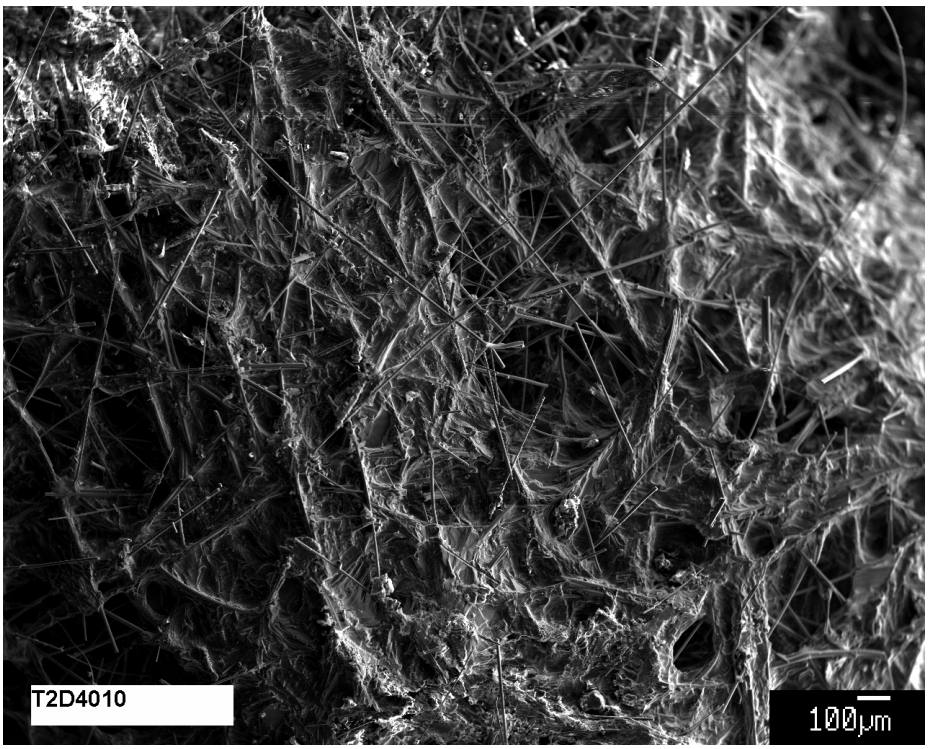


Figure J-10. Test-2 Day-4 SEM image, overview of dry sample #2 (T2D4010).

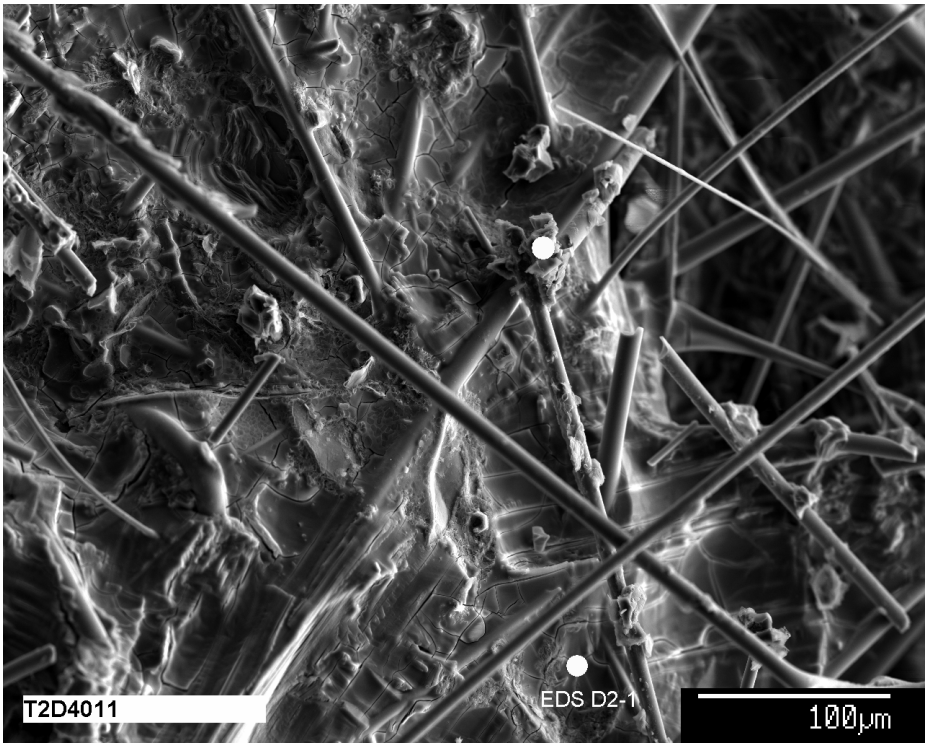


Figure J-11. Test-2 Day-4 SEM image of dry sample #2 deposits with crystals (T2D4011).

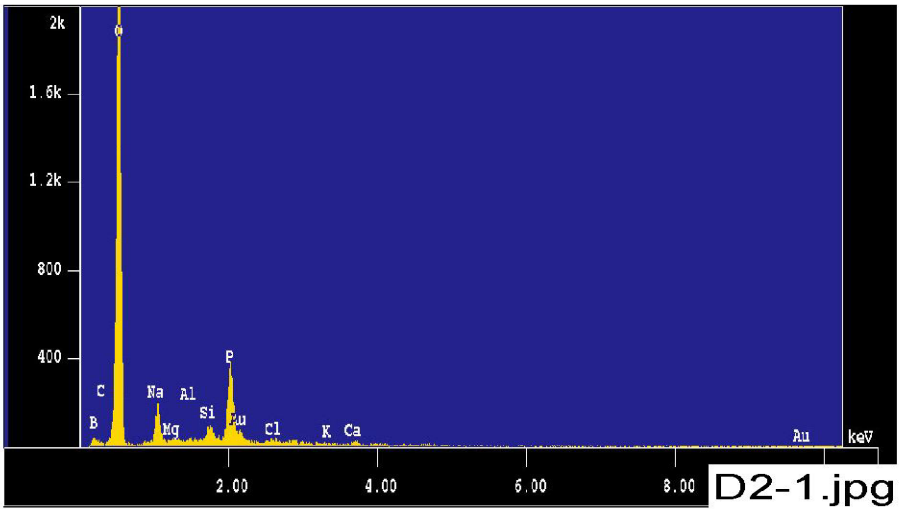


Figure J-12. Test-2 Day-4, dry sample #2 counting spectrum on the smooth deposits of image T2D4011 (Figure J-11) (D2-1).

The results from the chemical composition analysis for EDS D2-1 are given in Table J-1.

Table J-1. Chemical Composition for EDS D2-1

Feb 9 15:59 2005 /tmp/eds_pout.log Page 1

Group : NRC
Sample : T2D4 ID# : 1
Comment : Gunk filling between fibers
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 100.000 sec Aperture # : 1
Acc. Volt : 15.0 KV Probe Current : 1.030E-09 A
Stage Point : X=81.229 Y=64.853 Z=10.566
Acq. Date : Wed Feb 9 15:56:03 2005

Element	Mode	ROI(KeV)	K-ratio(%)	+/-	Net/Background
B K	Normal	0.00- 0.36	1.0120	0.0002	169 / 10
O K	Normal	0.25- 0.77	43.9423	0.0053	17656 / 26
Na K	Normal	0.83- 1.28	1.1193	0.0064	1414 / 26
Si K	Normal	1.50- 2.07	0.2683	0.0009	511 / 113
P K	Normal	1.75- 2.38	3.9584	0.0035	3798 / 64
Ca K	Normal	3.40- 4.30	0.2361	0.0040	238 / 14

Chi_square = 39.2421

Element	Mass%	Atomic%	ZAF	Z	A	F
B	15.604	22.5349	6.8867	1.1324	6.0813	1.0000
O	72.999	71.2365	0.7420	0.9755	0.7606	1.0000
Na	2.913	1.9780	1.1622	0.9805	1.1836	1.0014
Si	0.619	0.3442	1.0306	0.9774	1.0580	0.9966
P	7.357	3.7082	0.8301	1.1786	0.7043	1.0000
Ca	0.509	0.1982	0.9627	0.9931	0.9693	1.0001
Total	100.000	100.0000				

Normalization factor = 2.2389

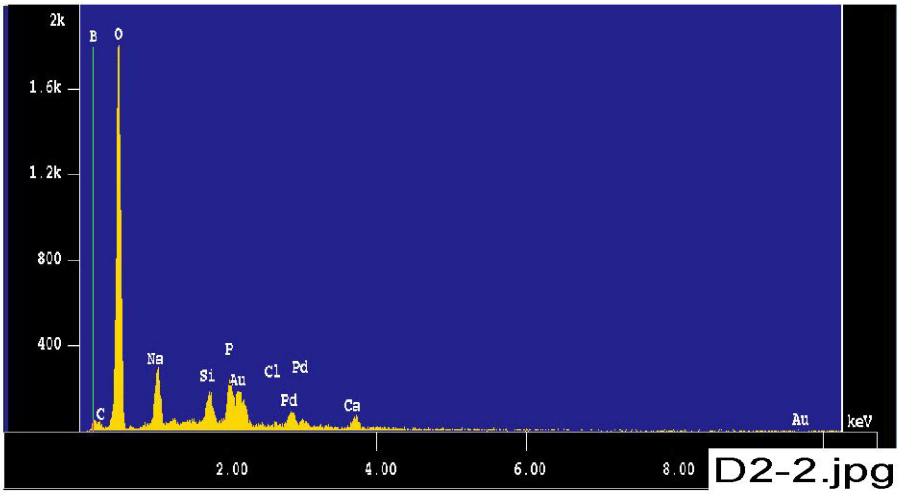


Figure J-13. Test-2 Day-4, dry sample #2 counting spectrum on the crystal mass on fibers of image T2D4011 (Figure J-11) (D2-2).

The results from the chemical composition analysis for EDS D2-2 are given in Table J-2.

Table J-2. Chemical Composition for EDS D2-2

Feb 9 16:07 2005 /tmp/eds_pout.log Page 1

Group : NRC
Sample : T2D4 ID# : 2
Comment : Crystals on fibers
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 100.000 sec Aperture # : 1
Acc. Volt : 15.0 KV Probe Current : 1.030E-09 A
Stage Point : X=81.182 Y=64.792 Z=10.566
Acq. Date : Wed Feb 9 16:03:32 2005

Element	Mode	ROI(KeV)	K-ratio(%)	+/-	Net/Background
B K	Normal	0.00- 0.36	0.9884	0.0003	165 / 9
O K	Normal	0.25- 0.77	37.3026	0.0050	14988 / 32
Na K	Normal	0.83- 1.28	1.7930	0.0083	2266 / 40
Si K	Normal	1.50- 2.07	0.6948	0.0011	1323 / 112
P K	Normal	1.75- 2.38	1.8701	0.0033	1794 / 102
Ca K	Normal	3.40- 4.30	0.7027	0.0056	708 / 18
Cl K	Normal	2.34- 3.06	0.3090	0.0008	329 / 31

Chi_square = 26.4409

Element	Mass%	Atomic%	ZAF	Z	A	F
B	16.075	23.2987	6.6100	1.1313	5.8430	1.0000
O	70.748	69.2911	0.7708	0.9745	0.7910	1.0000
Na	5.077	3.4601	1.1507	0.9795	1.1731	1.0015
Si	1.782	0.9942	1.0424	0.9763	1.0696	0.9982
P	3.881	1.9632	0.8433	1.1773	0.7166	0.9996
Ca	1.661	0.6492	0.9604	0.9919	0.9681	1.0001
Cl	0.777	0.3435	1.0221	1.0345	0.9888	0.9992
Total	100.000	100.0000				

Normalization factor = 2.4605

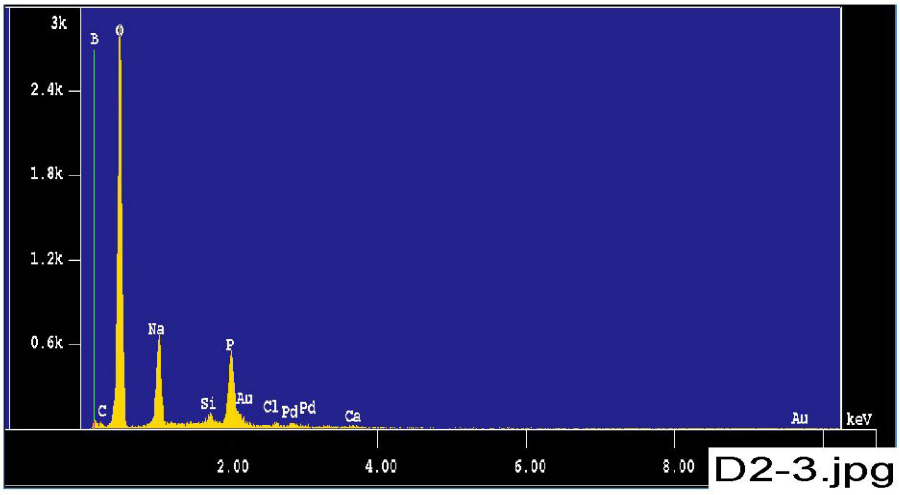


Figure J-14. Test-2 Day-4, dry sample #2 counting spectrum on the smooth cracked deposit on image T2D4011 (Figure J-11) (D2-3).

The results from the chemical composition analysis for EDS D2-3 are given in Table J-3.

Table J-3. Chemical Composition for EDS D2-3

Feb 9 16:19 2005 /tmp/eds_pout.log Page 1

Group : NRC
Sample : T2D4 ID# : 3
Comment : Cracked gunk
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 100.000 sec Aperture # : 1
Acc. Volt : 15.0 KV Probe Current : 1.027E-09 A
Stage Point : X=81.136 Y=64.586 Z=10.566
Acq. Date : Wed Feb 9 16:17:50 2005

Element	Mode	ROI(KeV)	K-ratio(%)	+/-	Net/Background
B K	Normal	0.00- 0.36	1.6161	0.0003	269 / 16
O K	Normal	0.25- 0.77	57.3942	0.0061	22993 / 42
Na K	Normal	0.83- 1.28	4.0848	0.0114	5146 / 33
Si K	Normal	1.50- 2.07	0.2639	0.0009	501 / 154
P K	Normal	1.75- 2.38	5.9164	0.0043	5660 / 76
Ca K	Normal	3.40- 4.30	0.0703	0.0042	71 / 22
Cl K	Normal	2.34- 3.06	0.3471	0.0008	368 / 34

Chi_square = 37.8736

Element	Mass%	Atomic%	ZAF	Z	A	F
B	17.669	25.5719	7.2292	1.1279	6.4095	1.0000
O	66.963	65.4859	0.7714	0.9716	0.7940	1.0000
Na	6.865	4.6718	1.1112	0.9766	1.1362	1.0014
Si	0.415	0.2309	1.0385	0.9734	1.0704	0.9966
P	7.444	3.7603	0.8319	1.1738	0.7089	0.9998
Ca	0.102	0.0399	0.9614	0.9890	0.9720	1.0001
Cl	0.542	0.2391	1.0323	1.0314	1.0009	1.0000
Total	100.000	100.0000				

Normalization factor = 1.5124

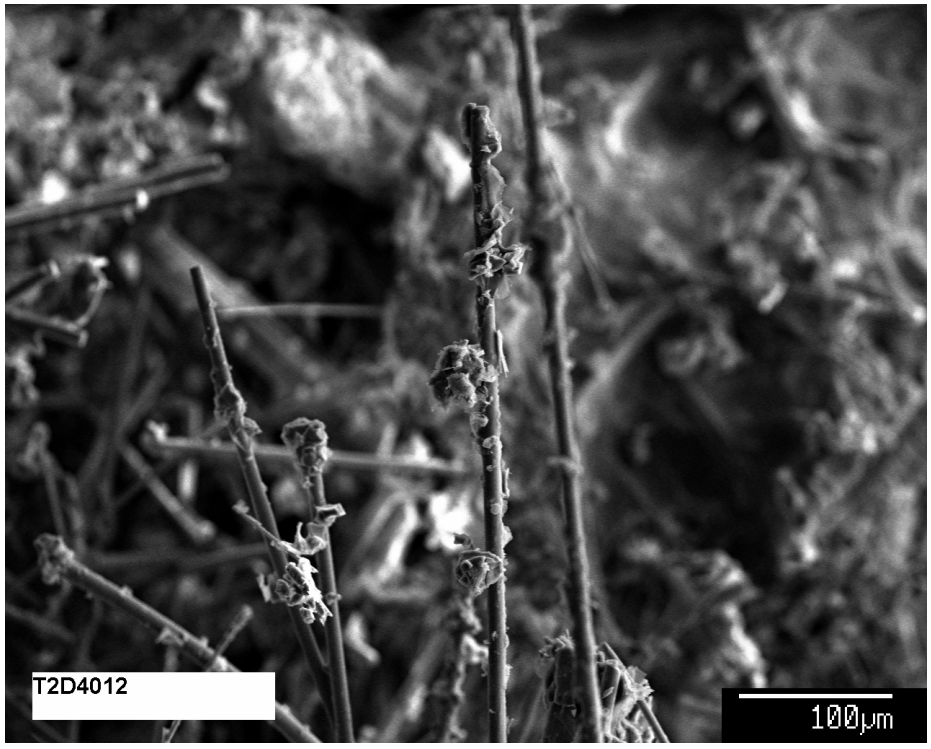


Figure J-15. Test-2 Day-4 SEM image of crystals on fibers of dry sample #2 at 200 × magnification (T2D4012).

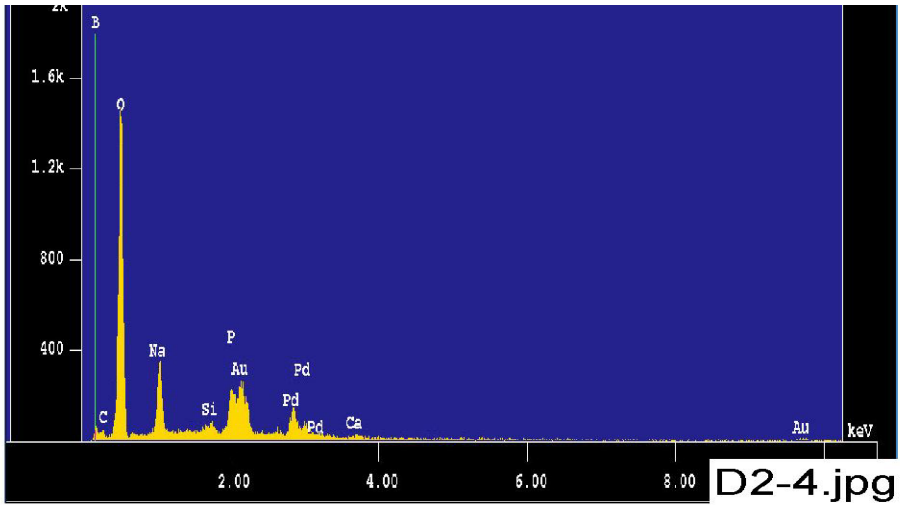


Figure J-16. Test-2 Day-4, dry sample #2 counting spectrum on the mass of crystals on fibers at the center of image T2D4012 (Figure J-12) but taken from an agglomeration at the tip of a fiber (D2-4).

The results from the chemical composition analysis for EDS D2-4 are given in Table J-4.

Table J-4. Chemical Composition for EDS D2-4

Feb 9 16:27 2005 /tmp/eds_pout.log Page 1

Group : NRC
Sample : T2D4 ID# : 4
Comment : Xtals on fibers
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 100.000 sec Aperture # : 1
Acc. Volt : 15.0 KV Probe Current : 1.025E-09 A
Stage Point : X=81.535 Y=63.667 Z=10.566
Acq. Date : Wed Feb 9 16:25:19 2005

Element	Mode	ROI(KeV)	K-ratio(%)	+/-	Net/Background
B K	Normal	0.00- 0.36	1.2483	0.0003	208 / 16
O K	Normal	0.25- 0.77	31.5962	0.0047	12633 / 34
Na K	Normal	0.83- 1.28	1.9840	0.0085	2495 / 36
Si K	Normal	1.50- 2.07	0.1494	0.0008	283 / 108
P K	Normal	1.75- 2.38	1.5144	0.0034	1446 / 68
Ca K	Normal	3.40- 4.30	0.1440	0.0043	144 / 23

Chi_square = 23.4498

Element	Mass%	Atomic%	ZAF	Z	A	F
B	21.555	30.0161	6.0487	1.1237	5.3829	1.0000
O	67.735	63.7363	0.7509	0.9681	0.7757	1.0000
Na	6.299	4.1245	1.1121	0.9732	1.1410	1.0015
Si	0.441	0.2365	1.0343	0.9702	1.0677	0.9984
P	3.580	1.7400	0.8280	1.1700	0.7077	1.0000
Ca	0.390	0.1467	0.9501	0.9862	0.9633	1.0001
Total	100.000	100.0000				

Normalization factor = 2.8548

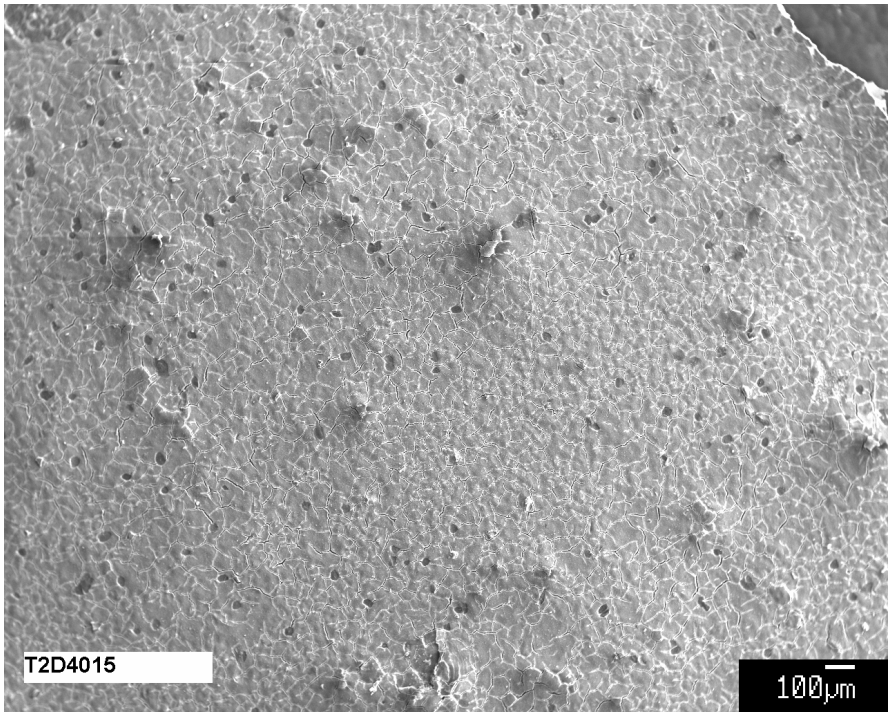


Figure J-17. Test-2 Day-4 SEM image, overview of the filtrate surface for the filter sample (T2D4015).

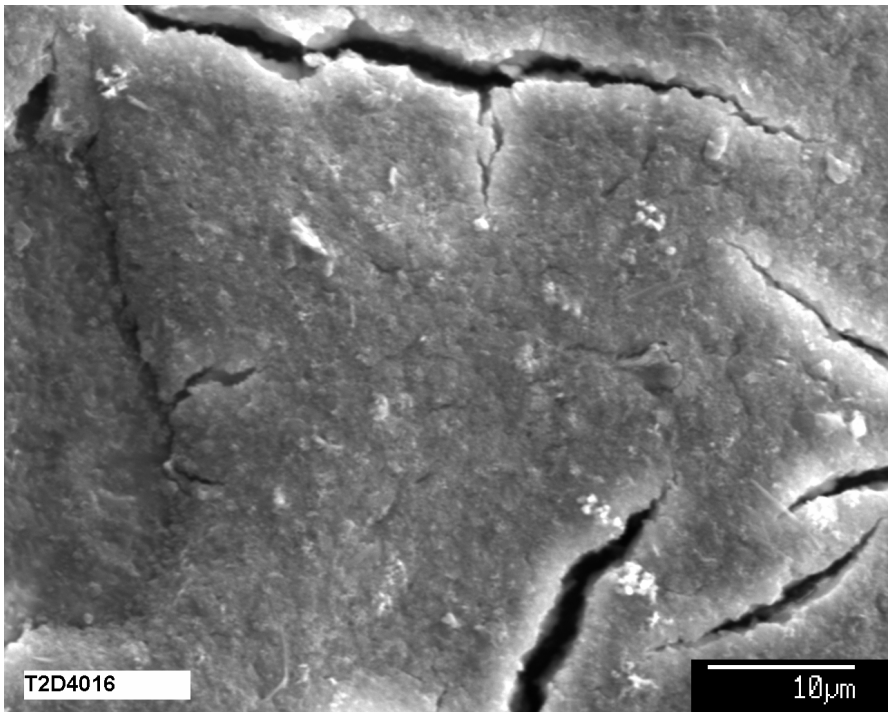


Figure J-18. Test-2 Day-4 SEM image of a close-up of filtrate for the filtrate sample, magnification is 2000 × (T2D4016).

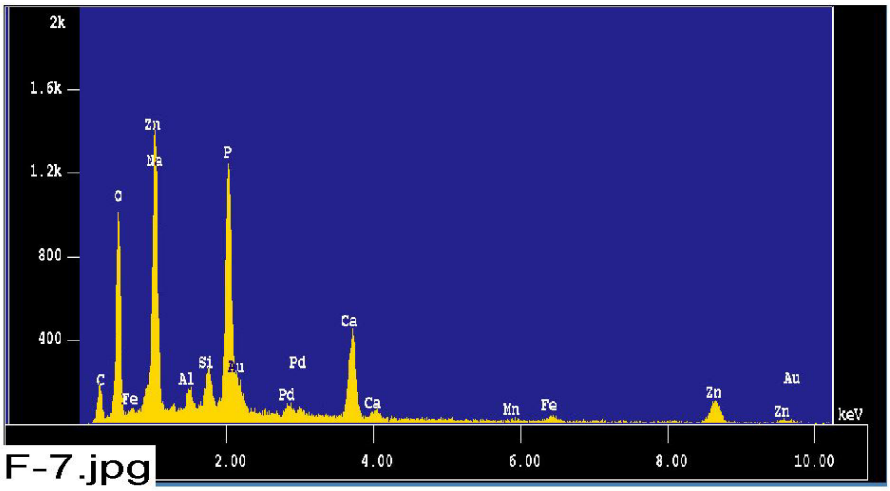


Figure J-19. Test-2 Day-4, filter sample counting spectrum for filtrate (F-7).

The results from the chemical composition analysis for EDS F-7 are given in Table J-5.

Table J-5. Chemical Composition for EDS F-7

Feb 9 17:04 2005 /tmp/eds_pout.log Page 1

Group : NRC
Sample : T2D4 ID# : 7
Comment : Filtrate
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 100.000 sec Aperture # : 1
Acc. Volt : 15.0 KV Probe Current : 1.151E-09 A
Stage Point : X=70.927 Y=64.105 Z=10.447
Acq. Date : Wed Feb 9 16:58:16 2005

Element	Mode	ROI(KeV)	K-ratio(%)	+/-	Net/Background
C K	Normal	0.09- 0.46	0.9733	0.0006	648 / 104
O K	Normal	0.25- 0.77	22.5680	0.0047	10133 / 118
Al K	Normal	1.26- 1.78	0.4614	0.0009	982 / 155
Si K	Normal	1.50- 2.07	0.7686	0.0014	1635 / 380
P K	Normal	1.75- 2.38	12.6137	0.0066	13524 / 196
Ca K	Normal	3.40- 4.30	4.7780	0.0122	5376 / 36
Fe K	Normal	6.04- 7.40	0.7955	0.0560	288 / 22
Zn K	Normal	8.22-10.03	13.8176	0.0110	1792 / 8
Mn K	Normal	5.53- 6.82	0.2228	0.0010	92 / 20
Na K	Normal	0.83- 1.28	0.4176	0.0235	590 / 93

Chi_square = 4.9155

Element	Mass%	Atomic%	ZAF	Z	A	F
C	7.802	14.9047	5.5330	0.9853	5.6158	1.0000
O	39.654	56.8718	1.2129	0.9394	1.2911	1.0000
Al	0.921	0.7829	1.3773	0.9502	1.4523	0.9980
Si	1.339	1.0937	1.2023	0.9383	1.2871	0.9955
P	16.843	12.4771	0.9217	1.1307	0.8154	0.9997
Ca	6.660	3.8131	0.9622	0.9486	1.0151	0.9993
Fe	1.061	0.4357	0.9202	0.9401	1.0059	0.9730
Zn	24.469	8.5891	1.2224	1.2235	0.9991	1.0000
Mn	0.375	0.1566	1.1615	1.1704	1.0083	0.9843
Na	0.877	0.8753	1.4496	0.9430	1.5352	1.0013
Total	100.000	100.0000				

Normalization factor = 1.4487

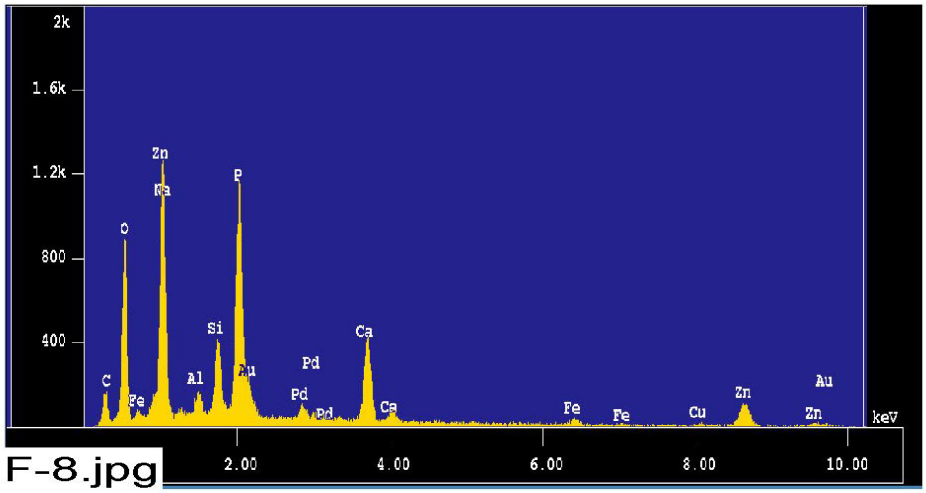


Figure J-20. Replicate Test-2 Day-4 filter sample counting spectrum for filtrate (F-8).

The results from the chemical composition analysis for EDS F-8 are given in Table J-6.

Table J-6. Chemical Composition for EDS F-8

Feb 9 17:12 2005 /tmp/eds_pout.log Page 1

Group : NRC
Sample : T2D4 ID# : 8
Comment : Filtrate
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
Live Time : 100.000 sec Aperture # : 1
Acc. Volt : 15.0 KV Probe Current : 1.140E-09 A
Stage Point : X=69.995 Y=63.989 Z=10.447
Acq. Date : Wed Feb 9 17:10:35 2005

Element	Mode	ROI(KeV)	K-ratio(%)	+/-	Net/Background
C K	Normal	0.09- 0.46	1.1312	0.0005	746 / 97
O K	Normal	0.25- 0.77	18.7823	0.0040	8352 / 116
Al K	Normal	1.26- 1.78	0.4612	0.0009	972 / 185
Si K	Normal	1.50- 2.07	1.4997	0.0016	3159 / 359
P K	Normal	1.75- 2.38	11.3019	0.0063	12002 / 232
Ca K	Normal	3.40- 4.30	4.7008	0.0119	5238 / 34
Fe K	Normal	6.04- 7.40	0.9343	0.0551	335 / 10
Zn K	Normal	8.22-10.03	14.3829	0.0110	1847 / 7
Na K	Normal	0.83- 1.28	0.3713	0.0223	519 / 71
Cu K	Normal	7.63- 9.27	0.7781	0.0041	132 / 10

Chi_square = 4.8053

Element	Mass%	Atomic%	ZAF	Z	A	F
C	9.451	18.4623	5.6231	0.9795	5.7408	1.0000
O	35.077	51.4416	1.2569	0.9339	1.3459	1.0000
Al	0.958	0.8327	1.3973	0.9445	1.4824	0.9980
Si	2.706	2.2607	1.2144	0.9326	1.3073	0.9960
P	15.710	11.9007	0.9356	1.1238	0.8327	0.9997
Ca	6.694	3.9187	0.9584	0.9426	1.0175	0.9993
Fe	1.265	0.5314	0.9111	0.9337	1.0064	0.9696
Zn	25.930	9.3071	1.2134	1.2144	0.9992	1.0000
Na	0.813	0.8294	1.4731	0.9374	1.5694	1.0013
Cu	1.396	0.5154	1.2074	1.2074	1.0000	1.0000
Total	100.000	100.0000				

Normalization factor = 1.4858

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